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(54) **Method for extracting sulphur from coals and lignatic materials**

(57) Coal to be desulphurised is first ground, and the resulting particles are placed in a container with trichloroethylene, tetrachloroethylene or other ethylenic compound, as a solvent, and the resulting mixture is heated to the boiling point of the compound. The mixture is maintained at the boiling point for a selected time interval, and the coal is then removed by filtration. The remaining filtrate is permitted to cool slowly to room temperature, and this slow cooling causes free sulphur which was removed from the coal to precipitate from the solution as monoclinic needles of high purity. Organic sulphur compounds, such as the mercaptans and thiophenes dissolved out of the coal remain in solution.

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SPECIFICATION

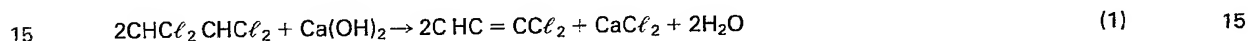
Method for extracting sulphur from coals and lignatic materials

5 *Background of the invention*

The alkenes are a class of unsaturated hydrocarbons containing one or more carbon-to-carbon double bonds. Ethylene [$\text{CH}_2 = \text{CH}_2$] is an example of an alkene. The process of the invention is predicated on the discovery that halogenated ethylene compounds dissolve free, binary, conjugated sulphur from bituminous coals and the like. The alkenes, on the other hand, have proven to be ineffective for this purpose.

- 10 The specific halogenated ethylene compound used in the process is the organic chlorine compound trichloroethylene, which is a known non-flammable solvent, and which is presently used to some extent for dry cleaning, metal degreasing, paint and varnish removal, and fats and oils extraction.

Trichloroethylene may be formulated by lime treatment in accordance with the following formula:



Trichloroethylene is a colorless, non-flammable, stable, heavy, mildly hazardous liquid at room temperature, and as stated above is presently used for metal degreasing and as an extraction solvent.

20 *Summary of the invention*

According to one aspect of the invention, there is provided a method for extracting sulphur from coal, and the like, comprising: providing a quantity of coal to be desulphurized; and treating the coal with an ethylenic compound as a solvent for extracting sulphur container in the coal.

Preferably, the coal is ground to a particulate form and the resulting coal particles mixed with the solvent.

- 25 The method conveniently includes the steps of heating the solvent and coal mixture to the boiling point of the solvent, removing the coal particles after a predetermined time interval, and then permitting the resulting solvent solution to cool to room temperature.

Preferably, the solvent and coal mixture are maintained at a temperature corresponding to the boiling point of the solvent for an interval of from 15-60 minutes. The solvent may be trichloroethylene or

- 30 tetrachloroethylene.

The method may further include the step of filtering out precipitated sulphur crystals from the solvent solution which form when the solvent solution is slowly cooled to room temperature. Dissolved organic sulfur compounds may thereafter be removed from the solvent solution after the solvent solution has been slowly cooled to room temperature.

- 35 The method further provides for the treatment of the solvent solution with Darco G60 to recover the solvent for re-use.

Detailed description of preferred embodiments of the invention

Specific example of the process of the invention:

- 40 A. Coal ground to particulate form is placed in a container with liquid trichloroethylene or tetrachloroethylene solvent in a ratio by weight of 1:1.5-2.0.

B. The mixture is then heated to the boiling point of trichloroethylene (86.7°C - 87°C) or tetrachloroethylene, and is held at the boiling point for from 15-60 minutes.

- 45 C. The solvent solution is then rapidly filtered from the coal, so that coal particles are recovered which are free from sulphur and from organic sulphur compounds.

D. The solvent solution is then slowly cooled to room temperature so as to recover sulphur of high purity.

- 50 As the solvent slowly cools to room temperature, sulphur nuclei form at the surface of the solvent with a Brownian oscillatory movement, and the nuclei rapidly grow to long monoclinic prisms to form homogenous crystals of pure sulphur. The pure sulphur may be removed from the solution by filtration. The organic sulphur compounds remain in the solution, and can be removed if desired, by any of a variety of known chemical techniques.

The solvent may then be treated with Darco G60 and re-used, if so desired.

- 55 Organic sulphur compounds, such as the mercaptans and thiophenes dissolved out of the coal remain in solution. The result is the disulphurization of the coal to enhance its heating capabilities, and also the production of sulphur of high purity as a precipitate which is free from tars, oils and organic sulphur compounds.

When the process is carried out on a continuous basis, counter-current commercial extraction may be employed at the boiling point of the solvent, and at pressures of from 1-3 atmospheres.

- 60 In carrying out the process of the invention with various sulphur bearing coals the following physical changes occurred which cannot be scientifically explained.

Example:

100 grams of coal (Wyoming Basin) were pulverized and treated with 20cc of trichloroethylene for 15 minutes at the boiling point. The following were observed.

5		<i>Before</i>	<i>After</i>	<i>Reduction</i>	5
	S	1.11%	0.98%	12%	
	Ash	9.50	8.90	17	
	Moisture	2.83	1.73	39	
10	BTU	13,326	13,600		10

Coal from another seam showed a sulphur reduction of 40% in 25 minutes treatment with the trichloroethylene at boiling point, with substantial reduction in ash content and a rise in BTU.

15 CLAIMS

1. A method for extracting sulphur from coal, and the like, comprising: providing a quantity of coal to be desulphurized; and treating the coal with an ethylenic compound as a solvent for extracting sulphur contained in the coal. 20
2. The method defined in claim 1, and which includes the step of grinding the coal to particulate form and mixing the resulting coal particles with the solvent.
3. The method defined in claim 2, and which comprises heating the solvent and coal mixture to the boiling point of the solvent, removing the coal particles after a predetermined time interval, and then 25 permitting the resulting solvent solution to cool to room temperature.
4. The method defined in claim 2, and which includes mixing the coal particles with the solvent in a ratio by weight of 1:1.5-2.0.
5. The method defined in claim 3, and which comprises maintaining the solvent and coal mixture at a temperature corresponding to the boiling point of the solvent for an interval of from 15-60 minutes.
- 30 6. The method defined in claim 3, and which includes the step of filtering out precipitated sulphur crystals from the solvent solution which form when the solvent solution is slowly cooled to room temperature.
7. The method defined in claim 6, and which includes the step of removing dissolved organic sulphur compounds from the solvent solution after the solvent solution has been slowly cooled to room 35 temperature.
8. The method defined in claim 7, and which includes the step of treating the solvent solution with Darco G60 to recover the solvent for re-use.
9. The method defined in claims 1 to 8, in which the solvent is trichloroethylene.
10. The method defined in claims 1 to 8, in which the solvent is tetrachloroethylene.
- 40 11. A method for extracting sulfur from coal substantially as hereinbefore specifically described with particular reference to the Examples. 40
12. Sulfur when produced by a process according to any one of claims 1 to 11.
13. Coal desulfurised by a process as claimed in any one of claims 1 to 11.